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# Effect of Electrolyte Concentration in Electrochemical Deposition on CO-NI-FE Film Composition

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## Abstract

A study on electrochemical deposition in a triple-component Co-Ni-Fe system from a chloride electrolyte solution with equal concentrations of Co, Ni, and Fe was performed. The concentrations used were 0.48; 0.083; 0.00625 mol/l, and the temperature was 70°. The relative content of the components in the film approached the composition of the electrolyte, when the concentration of each component was slightly decreased. The dependence of the composition of films on the current density is explained by concentration polarization.

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*Index terms*— Co-N-Fe films, chloride electrolyte, partial ion current balance, electrochemical deposition.

## 1 I. Introduction

Thin films of triple alloys consisting of the ferromagnetic metals Fe, Co and Ni have a high magnetization saturation, high magnetic permeability and low coercivity, and they are used in many areas, such as computers, read/write heads and microelectromechanical systems (MEMS). Electrochemical deposition is one of the most preferred methods for producing thin layers of alloy due to its simplicity, cost-effectiveness, versatility and relatively rapid deposition.

A characteristic feature of the electrochemical deposition of Co-Ni-Fe films is the discrepancy between the fraction of elements in the electrolyte and in the film, which makes it difficult to obtain a film with the desired electrophysical properties. This paper presents the results of a study on electrochemical deposition of a triple Co-Ni-Fe system from a chloride electrolyte solution with equivalent concentrations of Co, Ni, and Fe at 0.48, 0.083, and 0.00625 mol/l and a temperature of 70°C. The study of electrochemical deposition of Co-Ni-Fe alloys provides new insight into the mechanisms that determine the composition of the precipitated layers and their dependence on the composition of the electrolyte.

Finding the composition of the electrolyte to obtain the desired composition of the precipitated film is a very time-consuming task. The decision on the composition of the electrolyte depends on the introduction of additives into the electrolyte that improves the mechanical properties, adhesion and morphology of the films. The conducted studies of congruent electrochemical deposition of the triple Co-Ni-Fe system with equal concentrations of components in the electrolyte allow us to approach finding a solution to the electrolyte composition problem that determines the composition of precipitated films of complex composition.

## 2 II. Electrochemical Deposition of CO-NI-FE Films

For the deposition of Co-Ni-Fe films, a chloride electrolyte solution containing three components,  $\text{CoCl}_2 \cdot 6\text{H}_2\text{O}$ ,  $\text{FeCl}_2 \cdot 4\text{H}_2\text{O}$ , and  $\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$ , was used, with the components in a 1:1:1 molar ratio; three concentrations were tested: 0.48, 0.083, and 0.00625 mol/l [1]–[3]. Various additives were added to the electrolyte solution in the following concentrations:  $\text{H}_3\text{BO}_3$  -20 g/l,  $\text{C}_7\text{H}_4\text{NaNO}_3\text{S}_2\text{H}_2\text{O}$  -1.5 g/l,  $\text{HCl}$  -3 ml/l. The film from the specified electrolyte was deposited in a galvanic bath with a volume of 2 liters and a graphite anode. A vertically-oriented metallized silicon wafer was used as the cathode. The distance between the anode and the cathode was 8 cm.

Insoluble hydroxides were removed by filtration. An alloy film with a diameter of 8 cm was obtained on the metallized Ni surface of a 100 mm silicon wafer. The electrolyte was heated by a submersible heater to 70°C and

## 2 II. ELECTROCHEMICAL DEPOSITION OF CO-NI-FE FILMS

44 mixed with a magnetic stirrer. A constant current density of 3 to 40 mA/cm<sup>2</sup> was maintained in the deposition  
45 area on the silicon wafer.

46 As the concentration of CoCl<sub>2</sub>, NiCl<sub>2</sub>, and FeCl<sub>2</sub> salts in the electrolyte solution increased, the resistance  
47 of the solution decreased, and this resistance determined the amount of current passing between the anode and  
48 the cathode and the voltage that changed linearly with increasing current. At a current density of 16 mA/cm<sup>2</sup>  
49, the voltage between the anode and the cathode was 2 V, 3.5 V, and 5.5 V in the different electrolyte solutions.

50 With a difference in electrolyte concentrations of 77 times, the growth rate of Co-Ni-Fe films practically does  
51 not depend on the concentration of the electrolyte. The growth rate of Co-Ni-Fe films increases with increasing  
52 current density and half the values calculated according to Faraday's law, i.e. the cathode current output is 0.5.

53 1. Electrolytes with Co, Ni, and Fe concentrations of 0.48 mol/l [2] precipitated at a current density of 3.6  
54 mA/cm<sup>2</sup>.

55 The composition of the film was Co 32 Ni 61,5 Fe 6,5. At a low current density, the composition of the film was  
56 characterized by a high nickel content and low iron and content. When the current density of the Co-Ni-Fe film  
57 increased to 15 mA/cm<sup>2</sup>, the iron content rose to 41.3%, and at a current density of 20 mA/cm<sup>2</sup>, it remained  
58 approximately the same at 42.35%. When the current density of the Co-Ni-Fe film increased to 15 mA/cm<sup>2</sup>,  
59 the nickel content decreased to 7.9%, and at a current density of 20 mA/cm<sup>2</sup>, it remained at the same level of  
60 7.85%. The actual change in the iron and nickel content at a current density of 10 mA/cm<sup>2</sup> changed the relative  
61 cobalt content to 59%. At current densities of 15 and 20 mA/cm<sup>2</sup>, the cobalt content had similar values of  
62 49 and 48%. 2. Figure 2 shows the relative contents of Co, Ni, and Fe in the film during deposition from the  
63 electrolyte solution when the concentration of each component was 0.083 mol/l. There is a weak dependence on  
64 the current density at a value of more than 25 mA/cm<sup>2</sup>; in the Co-Ni-Fe film, the nickel content is 19 -22%,  
65 the iron content is 28 -33%, and the cobalt content has a value of 55 -47%. With a lower current density, the  
66 composition of the Co-Ni-Fe film varies greatly; the nickel content decreases from 57 to 19%, the iron content  
67 increases from 5 to 28%, and the cobalt content ranges from 42 -55 -48%. Comparison of the dependences on the  
68 current density of the composition of films obtained from a threecomponent solution of CoCl<sub>2</sub>, NiCl<sub>2</sub>, FeCl<sub>2</sub>  
69 in which the concentration of the components was either 0.48 mol/l or 0.083 mol/l showed that the dependencies  
70 were similar. Increasing the current density led to a decrease in the nickel content and an increase in the iron  
71 content, and the predominant cobalt deposition persisted. Their dependence of the composition of the films on  
72 the current density was stable within a certain area.

73 At a current of more than 25 mA/cm<sup>2</sup>, the ratio of metal concentrations in the film to the metal concentrations  
74 in the electrolyte was 1.44 for cobalt, 0.97 for iron, and 0.54 for nickel, which is almost the same as the values  
75 at an electrolyte concentration of 0.48 mol/l, but closer to the concentrations in the electrolyte. The relative  
76 contents of Co, Ni, and Fe in the film differ from the composition of the electrolyte and are highly dependent  
77 on the current density. It is not possible to control the deposition with the current density to obtain a film  
78 composition equal to the composition of the electrolyte.

79 3. Electrochemical deposition of Co-Ni-Fe films was performed in an electrolyte solution in which the Co, Ni,  
80 Fe chloride concentrations were all 0.00625 mol/l. The solution also contained 30% hydrochloric acid (0.3 ml/l),  
81 and the temperature was 70°C. Redeposition was carried out from the same electrolyte solution, but with the  
82 addition of saccharin and boric acid, the results are shown in Table 1. The results of the study on the composition  
83 of the films are shown in Figure ??.

84 Without electrolyte additives the compositions of Co-Ni-Fe films shows with markers connected by lines. The  
85 Co content is indicated by rectangular markers and a dashed line. The Ni content is indicated by triangular  
86 markers and a dotted line. The Fe content is indicated by round markers and a solid line. For the samples that  
87 contained additives to the electrolyte (saccharin at concentrations of 3 g/l (at 10 mA/cm<sup>2</sup>) and 1.5 g/l (at 10.7  
88 mA/cm<sup>2</sup>) and boric acid at a concentration of 20 g/l (at 11.4 mA/cm<sup>2</sup>)), the composition values are presented  
89 in the form of individual points. Arrows indicate changes in the composition of films for the selected current in  
90 electrolyte solutions containing the appropriate additives.

91 As seen in the figure, the compositions of the films depend on the current, and at a current density of 12.2  
92 mA/cm<sup>2</sup>, the compositions of the metals in the Co-Ni-Fe films are close to 33% molar content of CoCl<sub>2</sub>, NiCl<sub>2</sub>,  
93 FeCl<sub>2</sub> salts in the electrolyte, i.e., congruent electrochemical deposition of the triple-component Co-Ni-Fe  
94 alloy is observed.

95 Additives to the electrolyte change the composition of the films and disrupt the congruence of deposition in  
96 various ways. Saccharin increases the content of Co and Ni and reduces the content of Fe. With a decrease in  
97 the additive from 3 to 1.5 g/l, the change in composition decreases. Boric acid at 20 g/l increases the content of  
98 Co and Fe and reduces the content of Ni.

99 The simultaneous addition of saccharin (1.5 g/l) and boric acid (20 ml/l) increases the content of Co, slightly  
100 reduces the content of Ni and greatly reduces the content of Fe.

101 Fig. ??: Dependence on the current density  $J$  in the range of 10 -12.8 mA/cm<sup>2</sup> percentage of the components  
102 of films C(Co; Ni; Fe), obtained from an electrolyte with an equal 33% molar content of salts CoCl<sub>2</sub>, NiCl<sub>2</sub>,  
103 FeCl<sub>2</sub>, each with a concentration of 0.00625 mol/l.

104 The thickness of the concentrated films was measured using an MSA-500 microsystem analyzer. The study of  
105 the composition of the films was carried out using a PhilipsXL 40 energy dispersion X-ray microanalyzer.

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106 The dependences of the composition of Co-Ni-Fe films and their magnetic properties on the conditions of  
107 electrochemical deposition were studied.

108 Figure 4 shows the dependences of the voltage between the anode and cathode on the stabilized current  
109 densities of 5-20 mA/cm<sup>2</sup> at equal concentrations of CoCl<sub>2</sub>, NiCl<sub>2</sub>, and FeCl<sub>2</sub> salts in the electrolyte.

110 The voltage drop across the interelectrode space varies almost linearly with increasing current density from 5  
111 to 20 mA/cm<sup>2</sup>, i.e. the ohmic conductivity of the electrolyte determines the current in the operating mode. In  
112 the galvanostatic mode of electrochemical deposition from the chloride electrolyte with equal concentrations of  
113 each component, a decrease of the concentration leads to an increase of the voltage drop across the interelectrode  
114 space due to a decrease of the amount of ions in the electrolyte. The results, which were obtained using the  
115 electrochemical deposition from the chloride electrolyte with the concentration of each component, M: 0.0074  
116 (3); 0.08 (2); and 0.5 (1), the development of the procedure for the electrolyte preparation with the spectrum  
117 control, and the deposition at a temperature of 70°C enable one to obtain a low coercive force of Co-Ni-Fe films  
118 in a rather wide range of iron content in the film.

### 119 3 III. Magnetization and Coercive Force of Co-Ni-Fe Films

120 The magnetization and coercive force of the Co-Ni-Fe films were determined by the hysteresis loop of the magnetic  
121 field flux on the magnetic properties analyzer. The composition of the films on the plates was determined  
122 using an energy-dispersive X-ray microanalyzer. The results of measurements of magnetic parameters: specific  
123 magnetization B/h, coercive force H<sub>c</sub> and composition of films of triple alloy Co-Ni-Fe are presented in Figure 6.

### 124 4 IV. Discussion of the Results

125 The contents of the components in the film, which were fabricated by the electrochemical deposition from the  
126 three-component solutions of FeCl<sub>2</sub>, CoCl<sub>2</sub>, and NiCl<sub>2</sub> with equal concentrations of the components, do  
127 not correspond to the composition of the electrolyte. At a high current density, when cobalt and iron are  
128 predominantly deposited, and a fraction of nickel in the deposit is small, the dependence of the film composition  
129 on the current density at the cathode is stabilized.

130 Fe-Co-Ni films were deposited by the electrochemical method from a sulfate chloride electrolyte [4], containing,  
131 mol / l: NiSO<sub>4</sub> · 0.304, NiCl<sub>2</sub> · 0.084, CoSO<sub>4</sub> 0.1, FeSO<sub>4</sub> 0.036, H<sub>3</sub>BO<sub>3</sub> 20 g / l, 2 g / l stabilizer, 4  
132 g / l tartaric acid, 4 g / l bleach, 0.1 g / l wetting agent and tartaric acid additives. Optimum conditions for  
133 obtaining high-quality films of Fe 15.6-20.6 Co 43.8-61.9 Ni 22.5-40 current density 4 A / dm<sup>2</sup>, temperature 40  
134 °C, pH 2.3-3.2, tartaric acid concentration 8-12 g / l, molar Co<sup>2+</sup> / Ni<sup>2+</sup> ratio = 0.26-0.4. The composition of  
135 the film depends on the current density, electrolyte temperature, and pH. The ratio of the content of elements  
136 in the electrolyte and the film is observed -CRL for Fe, Co = 3, and for Ni = 0.5. Cobalt and iron precipitate  
137 with a concentration greater than in the electrolyte, and less nickel.

138 For electrochemical deposition of iron, the abnormal deposition is characteristic. Iron is deposited more  
139 intensively than cobalt and nickel. Cobalt is deposited more intensively than nickel. [4].

140 Estimating the deposition rate based on electrochemical potentials assumes a normal deposition of nickel, but  
141 there are many factors in the processes that determine metal deposition.

142 The concept of congruent electrochemical deposition determines the equality of the electrolyte and sludge  
143 compositions.

144 The choice of current density used to obtain the desired composition of films is widely used in  
145 electrochemical deposition. The mechanism of the influence of the cathode current density on the composition  
146 of the resulting alloys is not defined.

147 One can ask the following: why does the composition of the Co-Ni-Fe film in our experiment depend on the  
148 density of the current, and at a high current density, why does the composition cease to change?

149 Here are some of the specifics of the processes. The resistance of the electrolyte in the electrode space is crucial  
150 for the ion conduction of dissolved salts.

151 The total current of the ions discharged on the cathode is much smaller than the current that is set during  
152 the process. This difference is due to the large current of ions that do not participate in deposition.

153 The nickel ions deposited on the cathode are formed on the anode at a high concentration and have little  
154 mobility. Iron ions are formed on the anode at a low concentration and have mobility due to the many ions  
155 of nickel. Cobalt ions are formed on the anode at a medium concentration but have greater mobility than iron  
156 atoms. These are the ions that are involved in the electrochemical reaction occurring during metal deposition.  
157 It is possible that such ions are hydroxide species formed by the hydrolysis of CoClOH, NiClOH, and FeClOH.

158 Current leakage is a continuous process, and the discharge of positive ions on the cathode should be  
159 accompanied by the formation of positive ions on the anode. The rate of deposition practically does not depend  
160 on the concentration of salts but depends on the density of the current, i.e., the number of positive ions created  
161 on the anode and discharged on the cathode. The mobility of active ions varies according to the different natures  
162 of the salts.

## 5 V. Chemical and Electrochemical

Reactions in Electrolyte a) Hydrogen index of CoCl<sub>2</sub>, NiCl<sub>2</sub>, FeCl<sub>2</sub> solutions In solutions of CoCl<sub>2</sub>, NiCl<sub>2</sub>, FeCl<sub>2</sub>, a hydrogen pH was measured using a testo 206 meter [6]. The dependence of pH on the concentration of onecomponent solutions in the concentration range of 0.01 -1 mol / l is shown in Figure 7. At a concentration of 1 mol / l, dissolving NiCl<sub>2</sub> gives pH = 5.8, CoCl<sub>2</sub> gives pH = 4.7, FeCl<sub>2</sub> gives pH = 2.7. The pH value characterizes the ion balance: the concentration of hydrogen, hydroxyl, acidity or alkalinity of water. Consequently, the hydrolysis of ferric chloride reduces the concentration of hydrogen during dissociation of water the most and increases the amount of hydroxyl.

A change in pH during dilution of the solution is not monotonous, but there are local peaks and a change in slope in the dependence. This means that a decrease in the concentration of the impurity is accompanied by a change in the degree of ionization and the value of the charge of ions. Local peaks are located in the dependences at different salt concentrations: NiCl<sub>2</sub> -0.5 mol / l; CoCl<sub>2</sub> -0.03 mol / l; FeCl<sub>2</sub> -0.02 mol / l. To obtain the same level of salt ionization, it is necessary to have solutions of each of the salts or of the order of 1 mol / l, or 0.01 mol / l. Prior to the peaks, the dependences of pH on salt concentration are linear. After peaks, there is practically no dependence of pH on the concentration of salts of NiCl<sub>2</sub>, CoCl<sub>2</sub>, but for FeCl<sub>2</sub> there is a change, but at a slower rate than in the linear section.

## 6 b) Hydrolysis Reaction and Electrode Reactions

The experimental features of electrochemical deposition are described by a sequence of chemical and electrochemical reactions. The dissolution of cobalt, nickel and iron chlorides is accompanied by a hydrolysis reaction [5].  

$$\text{FeCl}_2 + \text{H}_2\text{O} \rightleftharpoons \text{FeClOH} + \text{H}^+ + \text{Cl}^-$$

$$\text{NiCl}_2 + \text{H}_2\text{O} \rightleftharpoons \text{NiClOH} + \text{H}^+ + \text{Cl}^-$$

$$\text{CoCl}_2 + \text{H}_2\text{O} \rightleftharpoons \text{CoClOH} + \text{H}^+ + \text{Cl}^-$$

On the anode, there is an electrochemical reaction, and in accordance with the size of the current and the potentials of ionization, positive ions with different concentrations are formed. At the nickel anode, chlorine dissolves the electrode when produced.

Graphite anodes are successfully used in the electrolysis of chloride salt solutions, and the anode potential at them is low. The products of the destruction of graphite anodes do not contaminate the cathode metal. On graphite anodes, chlorine is released.  

$$\text{Fe}^{2+} + \text{H}^+ + \text{Cl}_2 \rightarrow \text{Fe}^{3+} + \text{H}^+ + \text{Cl}_2$$

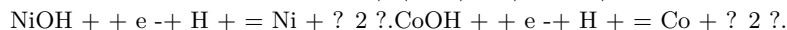
$$\text{Ni}^{2+} + \text{H}^+ + \text{Cl}_2 \rightarrow \text{Ni}^{3+} + \text{H}^+ + \text{Cl}_2$$

$$\text{Co}^{2+} + \text{H}^+ + \text{Cl}_2 \rightarrow \text{Co}^{3+} + \text{H}^+ + \text{Cl}_2$$

Under the influence of the electric field in the electrolyte, there is a drift in the positive ions of the metal hydroxides from the anode to the cathode at a speed determined by the magnitude of the mobility of the ions and the tension of the electric field.

On the cathode, there is an electrochemical reaction of metal discharge from hydroxide species and the formation of water molecules.  

$$\text{FeOH} + \text{e}^- + \text{H}^+ = \text{Fe} + \text{H}_2\text{O}$$



On the cathode, there is an electrochemical reaction of metal discharge from hydroxide species and the formation of water molecules.

## 7 c) Concentration Polarization

The change in the electrode potential due to a change in the concentration of reagents in the electrode space during the passage of current is called concentration polarization [10]. Electrochemical reactions on the electrodes lead to a significant change in the concentrations of substances due to the slow diffusion of reagents or the removal of reaction products. The difference in the diffusion coefficients or mobility of the ions of the electrolyte component determines the balance of partial ionic currents and the content of the components in the film from the current density.

Based on the concept of concentration polarization, our results on the dependence of the concentration of the components of the triple alloy Co-Ni-Fe on the current density can be interpreted taking into account the dependence of the mobility of the ions, and not the potential of the electrode. Polarization of the electrode -the change in its potential has the same value for the ions of the three metals, and the mobility of show with a dynamic change in the potential of the electrode.

It has been experimentally established [11] that an area with an increased pH value is formed near the cathode (Figure 8). An increase in pH to a value of 7 in the region of 0.5 mm near the cathode indicates the release of water during a cathode electrochemical reaction. The greater the current density, the stronger the deviation of the composition of the electrolyte near the cathode from a uniform homogeneous one corresponding to the thermodynamic equilibrium occurs.

The ions of the electrolyte component have different diffusion coefficients during the diffusion mechanism of mass transfer and mobility values during drift of ions in an electric field, which determines the different dependence of the content of components in the film on the current density, which creates a layer of concentration polarization that limits the mass transfer of electroactive ions to the electrode. The formation of a layer with a high pH value in the rolling region of the electrolyte explains the dependence of the composition of the precipitate on the mixing of the electrolyte, which violates the layer of concentration polarization.

The thickness of the layer deposited by the electrolyte during an electrochemical reaction -SISEER is fairly

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222 large, on the order of a millimeter. A layer of this thickness has a significant effect on the mass transfer of ions  
223 to the electrodes.

224 The formation of a layer with a changed composition in the cathode region of the electrolyte explains the  
225 dependence of the composition of the sediment on the mixing of the electrolyte that destroys the concentration  
226 polarization layer.

227 The lower the concentration of the electrolyte is, the smaller the deviation of the composition of the films from  
228 the equilibrium state and the closer the composition of the films to the composition of the electrolyte. Therefore,  
229 congruent electrochemical deposition of Co-Ni-Fe alloy can be obtained only when the concentrations of the main  
230 components of the electrolyte are low and when boric acid and saccharin are not added.

231 The flow of ionic current disrupts the thermodynamic equilibrium in the electrolyte due to the release of the  
232 products of electrochemical reactions in the electrode regions, and the mixing of the electrolyte with mechanical or  
233 magnetic stirrers, when exposed to an ultrasonic field, during rotation or reciprocating movement of the cathode,  
234 erodes the electrode layers and evens out the distribution of salts in the electrolyte.

235 With a dynamic change in voltage at a speed of 10 mV / s and control of the current flowing through the  
236 electrode, the dynamic nature of the behavior of ion currents is manifested [12] (Figure 9). The presence of  
237 current peaks in the dynamic mode of voltage change is associated with the kinetic properties of the ions. The  
238 positive ions formed on the anode when approaching the cathode change the potential near the working electrode  
239 and reduce the current of the electrode. The difference in the mobility of cobalt, iron and nickel ions determines  
240 the temporal and amplitude change in the electrode current. The presence of current peaks in the dynamic mode  
241 of voltage change is associated with the kinetic properties of the ions. The positive ions formed on the anode  
242 when approaching the cathode change the potential near the working electrode and reduce the current of the  
243 electrode. The difference in the mobility of cobalt, iron and nickel ions determines the temporal and amplitude  
244 change in the electrode current.

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246 products of electrochemical reactions in the electrode regions, and the mixing of the electrolyte with mechanical or  
247 magnetic stirrers, when exposed to an ultrasonic field, during rotation or reciprocating movement of the cathode,  
248 erodes the electrode layers and evens out the distribution of salts in the electrolyte.

249 With a low cathode current density and a high concentration of salts in SISEER, a low concentration of  
250 residues of molecules arises, from which metal atoms are released and which limit the flow of ionic currents of all  
251 three metals. The composition of the film is determined by partial ion currents in accordance with the mobility  
252 of ions in the electrolyte with the initial concentration of components. The increase in current density leads to  
253 an increase in the content of molecule residues after the electrochemical reaction of metal release on the cathode.  
254 The contribution of partial currents of metal ions depends on the composition of the concentration polarization  
255 layer. There is a limitation in the deposited film of the nickel concentration alloy, which has a small amount  
256 of mobility. Iron-containing ions have more mobility in the concentration polarization layer. The concentration  
257 of iron in the film increases. The mobility of cobalt-containing ions in SISEER is high, which leads to a strong  
258 increase in the relative content of cobalt in the film.

259 Stirring the salt solution disrupts SISEER. Carrying out the process of electrochemical precipitation without  
260 mixing reduces the nickel content and increases the iron content in the film, since there is no violation of CISER.  
261 With a high cathode current density and a low concentration of salts in the electrolyte region near the cathode,  
262 the flow of ionic currents of all three metals is not limited. As a result, congruent deposition of the Co-Ni-Fe  
263 alloy film is observed.

264 Establishes [13] that at a given speed of rotation of the cathode, it is possible to select the density of the  
265 deposition current to obtain a given film composition at a different ratio of nickel and iron in the electrolyte.  
266 Electrochemical precipitation of Ni-Fe alloys with a wide spectrum of composition was carried out from a sulfate-  
267 chloride electrolyte containing Ni<sup>2+</sup> / Fe<sup>2+</sup> ions (nickel sulfate -iron chloride) in proportions of 5: 1, 10: 1, 15:  
268 1, 20: 1, 25: 1, boric and ascorbic acids, saccharin, sodium lauryl sulfate (Figure 10). The processes were carried  
269 out at current densities (20 -160 mA/cm<sup>2</sup>), room temperature 23°C, pH = 3.0. The cathode was a platinum disk  
270 moving at a speed of 0.0005 -0.008 cm / s. The change in cathode speed and current density made it possible,  
271 as shown in Figure 10, for a given ratio of nickel and iron 10: 1, 15: 1, 20: 1, 25: 1 in the electrolyte to obtain  
272 the composition of the permalloy film Ni 81 Fe 19 .

273 In our experiment with electrolyte stirring, according to the terminology adopted in the book [14], the effect of  
274 diffusion on the rate of electrochemical processes is not basic. The transport of electroactive ions is determined  
275 by the migration of ions at a high current of the background electrolyte. It should be noted that in the book  
276 it is believed that the voltage drop in the electrolyte occurs near the electrodes, and the field strength in the  
277 electrolyte is very small, which contradicts the continuity of the current.

278 Based on the experimental results of obtaining Co-Ni-Fe films, a mechanism of electrochemical deposition  
279 based on the phenomena of salt hydrolysis, discharge of positive ions at the anode, drift of ions in the volume of  
280 the electrolyte and discharge of negative ions at the cathode with precipitation of metals and the formation of  
281 SISEER [15], has been proposed.

282 Experimental data show a difference between the deposition rate of the three components of the Co-Ni-Fe alloy  
283 and the metal content in the electrolyte. The determining factor is the equality of the charges of the ions that  
284 form the partial currents of the electroactive ions. For the Triple Alloy Co-Ni-Fe, such conditions are met at an

## 8 VI. CONCLUSION

285 electrolyte component concentration of 0.06 M/L and a current density of 12 mA/cm<sup>2</sup>, which gives congruent  
286 deposition.

### 287 8 VI. Conclusion

288 The deposition of Co-Ni-Fe films was performed in a chloride electrolyte with a ratio of  $C_{Co} : C_{Ni} : C_{Fe} =$   
289 1: 1: 1, and technology for preparing an electrolyte via filtration and performing deposition at a temperature  
290 of 70°C was developed. It was found that the desired 1:1:1 ratio of concentrations of the metals in the film was  
291 achieved at a current density of 12.5 mA/cm<sup>2</sup> and a metal chloride concentration of 0.00625 M/L. Co-Ni-Fe  
292 films are obtained reproducibly with minimal mechanical stresses and with good adhesion to the nickel sublayer  
293 during electrochemical deposition. The mechanistic study of the electrochemical deposition of a Co-Ni-Fe alloy as  
294 the electrolyte concentration changes resulted in congruent deposition of the three-component alloy. The use of  
295 congruent electrochemical deposition will simplify the choice of electrolyte composition for obtaining films with a  
complex composition. A review of the literature shows that none of the researchers used this method of research.

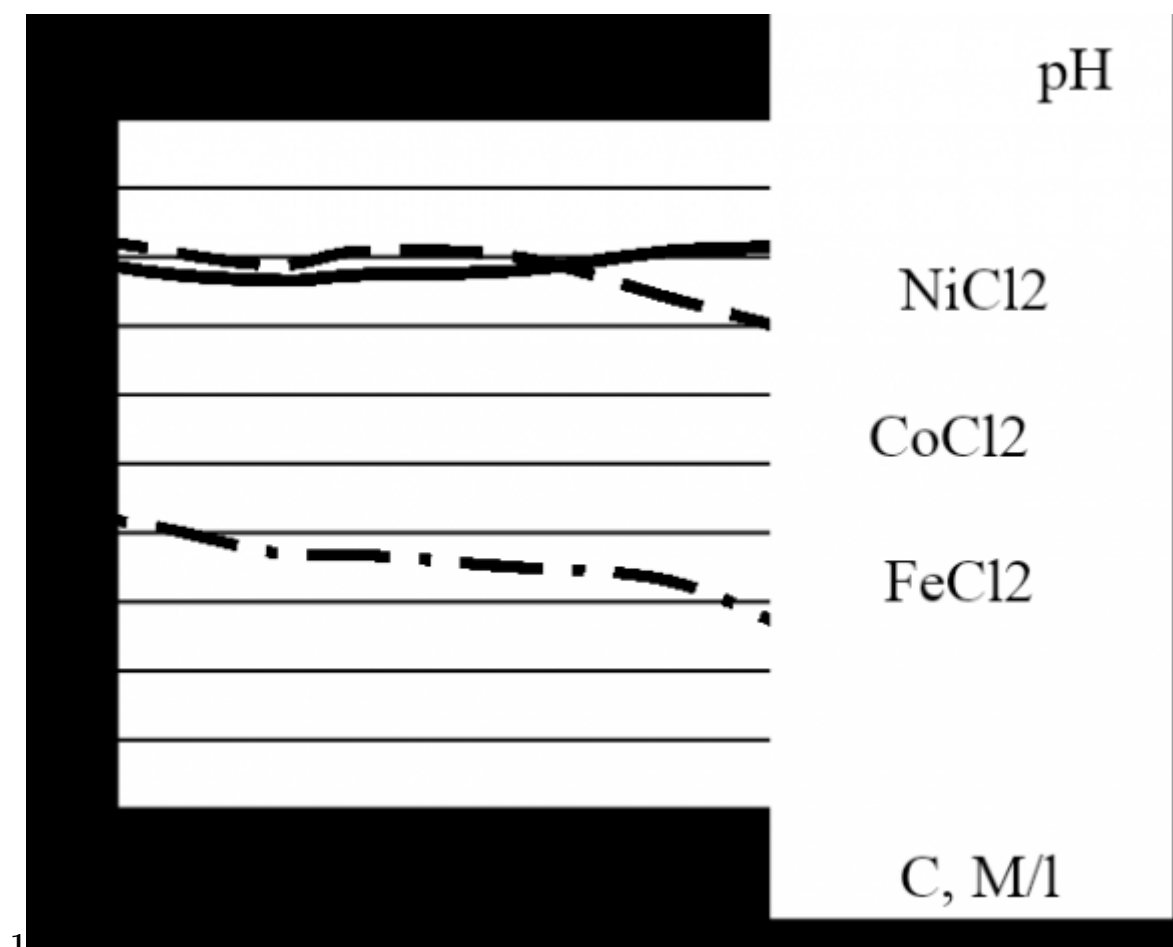


Figure 1: Fig. 1 :

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297

1

H and composition of Co-Ni-Fe films after electrochemical deposition in which each component had a concentration of 0.00625 mol/l. The solution also contained added boric acid and saccharin. Process parameters: pH of the electrolyte, voltage between the anode and cathode U, electric current I, deposition time t, film growth rate V.

# Ph	U, V	I, Ma	T, Min	H, $\mu$	V, Nm/Min	Co, %	Ni, %	Fe, %	Electrolyte
1	2,62	6,8			500 30	4,34	145	42	13,5 44
2	2,25	5,8			500 30	3,19	106	34	35 31 Saccharin 3 g/l
3	2,8	8,1			570 30	4,36	177	51	20 30
4	2,2	8,55			570 30	5,31	145	45,3	17,8 36,9 boric acid 20 ml/l
5	2,35	7,75			534 30	4,5	150	44,5	24 31,5 Saccharin 1,5 g/l
6	2,7	10,4			610 10	1,9	190	35,9	29,7 33,6
7	2,55	8,3			610 10	2,8	88	50,6	13,7 35,7 Saccharin 1,5 g/l boric acid 20 ml/l

Figure 2: Table 1 :





- 298 [Kuru et al.] , C H Kuru , H Kockar , O Demirbas , M Alper . 10.1007/s10854-015-2943-1. Characterizations of  
299 electrodeposited NiCoFe
- 300 [Yu and Zangari ()] , Gamburg D Yu , G Zangari . 2011. New York: Springer.
- 301 [ Journal of Materials Science Materials ()] , *Journal of Materials Science Materials* 2015. 26 (6) p. 4046.
- 302 [Yanai ()] *Electroplated Fe-Co-Ni films prepared from deep-eutectic-solvent-based plating baths// AIP Advances*,  
303 T Yanai . 2016. 6 p. 55917.
- 304 [Zech et al. ()] *Anomalous Codeposition of Iron Group Metals I. Experimental Results//Journal of The Electro-*  
305 *chemical Society*, N Zech , E J Podlaha , D Landolt . 1999. 146 p. .
- 306 [Leith et al.] ‘Characterization of Ni<sub>x</sub>Fe<sub>1-x</sub> (0.1<x<0.95) Electrodeposition from a Family of Sulfamate-Chloride  
307 Electrolytes’. S D Leith , Ramli Sh , D T Schwartz . *Journal of the Electrochemical Society* 1999 146 p. 1431.
- 308 [Gong et al.] ‘Composition gradients and magnetic properties of 5-100 nm thin CoNiFe films obtained by  
309 electrodeposition’. J Gong , Riemer , Morrone , Venkatasamy , I Kautzky , Tabakovic . *Journal of the*  
310 *Electrochemical Society* 2012 (7) p. .
- 311 [Tobakovic et al. ()] ‘Composition, structure, stress, and coercivity of electrodeposited soft magnetic CoNiFe  
312 films’. I Tobakovic , V Inturi , S Riemer . *Journal of The Electrochemical Society* 2002. 149 (1) p. .
- 313 [Tikhonov et al. (2021)] ‘Concentration dependence of electrodeposition of Co-Ni-Fe alloy//Eurasian Scientific  
314 Association, Results of Science in Theory and Practice’. R D Tikhonov , A A Cheremisinov , M R Tikhonov .  
315 [www.esa-conference.ru](http://www.esa-conference.ru) *82nd International Scientific Conference of the Eurasian Scientific Association*,  
316 (Moscow) 2021. December 24-25, 2021. 82 p. .
- 317 [Tikhonov ()] *Congruent electrochemical deposition of NiFe alloy*, Robert Tikhonov . Lambert Academic  
318 Publishing, 2019, P. 193.
- 319 [Damaskin et al. ()] B B Damaskin , O A Petriy , G A Cirlina . *Electrochemistry. Textbook for universities. 3-e*  
320 *ed., SPb.; M.; Krasnodar: Lan*, 2015. 672.
- 321 [Jian ()] *Effect of boron/ phosphorus containing additives on electrodeposited CoNiFe soft magnetic thin*  
322 *films//Transactions of Nonferrous Metals Society of China*, Li Jian , - . 2013. 23 p. 674.
- 323 [Phua et al. ()] ‘Effect of Ni concentration on microstructure, magnetic and microwave properties of electrode-  
324 posited NiCoFe films’. L X Phua , N N Phuoc , C K Ong . *Journal of Alloys and Compounds* 2012. 543 p.  
325 .
- 326 [Yun and Jiang ()] *Effects of additives on magnetic properties of electroplated CoNiFe films// Master’s Theses*,  
327 Tiffany Yun , Wen Jiang . 2008. San Jose State University
- 328 [Osaka et al. ()] ‘Effects of saccharin and thiourea on sulfur inclusion and coercivity of electroplated soft magnetic  
329 CoNiFe film’. T Osaka , T Sawaguchi , F Mizutani , T Yokoshima , M Takai , Y Okinaka . *Journal of The*  
330 *Electrochemical Society* 1999. p. .
- 331 [Eguchi et al.] K Eguchi , K Azuma , T Akiyoshi , H Fukunaga . *DC/Pulse Plating of Fe-Ni-Co*  
332 *Films//Conference: 2016 International Conference of Asian Union of Magnetism Societies (ICAUMS)*,
- 333 [Long et al.] ‘Electrochemical Deposition of Fe-Co-Ni Samples with Different Co Contents and Characterization  
334 of Their Microstructural and Magnetic Properties//Materials Science’. V C Long , U Saraç , M C Baykul , L D  
335 Trong , ??lu , D N Trong . 10.3390/coatings12030346. <https://doi.org/10.3390/coatings12030346>  
336 *Coatings* 2022 (3) p. 346.
- 337 [Cesiulis et al. ()] *Electrodeposition of Iron-Group Alloys into Nanostructured Oxide Membranes: Synthetic*  
338 *Challenges and Properties//Current Nanoscience*, H Cesiulis , N Tsyntaru , E J Podlaha , D Li , J Sort .  
339 2018. 14 p. .
- 340 [Park et al. ()] *Electrodeposition of low-stress high magnetic moment Fe-rich FeCoNi thin films//Electrochimica*  
341 *acta*, D Y Park , B Y Yoo , S Kelcher , N V Myung . 2006. 51 p. .
- 342 [Rohan et al. ()] ‘Electroless thin film CoNiFe-B alloys for integrated magnetics on Si’. J F Rohan , B M Ahern  
343 , K Reynolds , S Crowley , D A Healy , F M F Rhen , S Roy . *Electrochimica Acta* 2009. 54 (6) p. 1851.
- 344 [Yanai ()] ‘Electroplated Fe-Co-Ni films prepared in ammonium-chloride-based plating baths’. T Yanai . *AIP*  
345 *ADVANCES* 2018. 8 (056127) p. .
- 346 [Li ()] *Fabrication of Fe-Ni-Co Nanowires by Electrodeposition Coupled with Hydrogen Evolution Reaction*  
347 *and Electrochemical Detection of Pyocyanin//A*, D Li . 2018. Boston, Massachusetts. Thesis of Doctor  
348 Northeastern University
- 349 [Tikhonov] ‘Features of electrochemical deposition of films of the triple system of the CoNiFe’. R D Tikhonov .  
350 *European Journal of Engineering Research and Science* 2021 (2) p. .
- 351 [Perez et al. ()] ‘Magnetic properties of CoNiFe alloys electrodeposited under potential and current control  
352 conditions’. L Perez , K Attenborough , J De Boeck , J P Celis , C Aroca , P Sánchez , E López , M C  
353 Sánchez . *J. Magn. Magn. Mater* 2002. (1) p. .

- 354 [Kim et al. ()] *Magnetic properties of nanocrystalline iron group thin film alloys electrodeposited from sulfate and*  
355 *chloride baths//Electrochimica Acta*, D Kim , D Y Park , B Y Yoo , P T A Sumodjo , N V Myung . 2003. 48  
356 p. .
- 357 [Romankov et al. ()] ‘Mechanical intermixing of elements and selforganization of (FeNi) and (CoFeNi) nanos-  
358 tructured composite layers on a Ti sheet under ball collisions’. S Romankov , Y C Park , I V Shchetinin .  
359 *Journal of Alloys and Compounds* 2015. 653 p. .
- 360 [Nakano et al. ()] ‘Mechanism of Anomalous Type Electrodeposition of Fe-Ni Alloys from Sulfate Solu-  
361 tions//The’. H Nakano , M Matsuno , S Oue , M Yano , Kobayashi Sh , H Fukushima . *Materials Transactions*  
362 2004. Japan Institute of Metals. 45 (11) p. .
- 363 [Tikhonov] ‘Normal Electrochemical Deposition NiFe: Experiments and Theory’. R D Tikhonov . *European*  
364 *Journal of Engineering Research and Science* 2020 (8) p. .
- 365 [P’eter et al. ()] ‘On the composition depth profile of electrodeposited Fe-Co-Ni alloys//Chemistry’. L P’eter , A  
366 Csik , K Vad , E Toth-Kadar , A Pekker , G Molnár . *Electrochimica Acta* 2010. 55 (16) p. .
- 367 [Tabakovic et al. ()] ‘Organic additives in the electrochemical preparation of soft magnetic CoNiFe films’. I  
368 Tabakovic , S Riemer , V Inturi , P Jallen , A Thayer . *Journal of The Electrochemical Society* 2000.  
369 147 (1) p. .
- 370 [Kourov et al. ()] ‘Peculiar features of physical properties of the rapid quenched AlCrFeCoNiCu high-entropy  
371 alloy’. N I Kourov , V G Pushin , A V Korolev , Yu V Knyazev , M V Ivchenko , Yu M Ustyugov . *Journal*  
372 *of Alloys and Compounds* 2015. 636 p. .
- 373 [Hanafi et al. ()] *Potentiostatic electrodeposition of Co-Ni-Fe thin films from sulfate medium//Journal of*  
374 *Chemical Technology and Metallurgy*, I Hanafi , A R Daud , Radiman Sh . 2016. 51 p. .
- 375 [Yang ()] ‘Preparation of Fe-Co-Ni ternary alloys with electrodeposition’. Y Yang . *Int. J. Electrochem. Sci* 2015.  
376 10 p. .
- 377 [Tabakovic and Venkatasamy ()] ‘Preparation of metastable CoFeNi alloys with ultra-high magnetic saturation  
378 ( $B_s = 2.4-2.59$  T) by reverse pulse electrodeposition’. I Tabakovic , V Venkatasamy . *Journal of Magnetism*  
379 *and Magnetic Materials* 2018. 452 p. .
- 380 [Kayani and Riaz ()] ‘Sh. Naseem, Structural and magnetic properties of FeCoNi thin films//Indian J’. Z N  
381 Kayani , S Riaz . *Physics* 2014. 88 (2) p. .
- 382 [Huang and Podlaha ()] ‘Simulation of Pulsed Electrodeposition for Giant Magnetoresistance FeCoNiCu/Cu  
383 Multilayers’. Q Huang , E J Podlaha . *Journal Electrochemical Society* 2004. 151 (2) p. .
- 384 [Sundaram et al. ()] *Structural and magnetic properties of high magnetic moment electroplated CoNiFe thin*  
385 *films//Ionics*, K Sundaram , V Dhanasekaran , T Mahalingam . 2011. 17 p. .
- 386 [Romankov et al. ()] ‘Structural transformations in (CoFeNi)/Ti nanocomposite systems during prolonged  
387 heating’. S Romankov , Y C Park , I V Shchetinin . *Journal of Alloys and Compounds* 2018. 745 p. 44.
- 388 [Valko and Gurtovoy ()] *Structure and properties of coatings Co?Ni?Fe, electrolytically besieged by X-ray*  
389 *radiation//Fisika tverdogo tela*, N G Valko , W G Gurtovoy . 2013. 55 p. .
- 390 [Koda et al. ()] ‘Template-Assisted Electrodeposition of Fe-Ni-Co Nanowires: Effects of Electrolyte pH and  
391 Sodium Lauryl Sulfate’. T K Koda , K Eguchi , K Takashima . *Effect of Ammonium Chloride in Plating*  
392 *Baths on Soft Magnetic Properties of Electroplated Fe-Ni Films// IEEE Transactions on Magnetism*, E Li,  
393 Podlaha (ed.) 2017. 2017 p. D843. (PP)
- 394 [Tikhonov et al.] *The magnetic properties of the Co-Ni-Fe films were obtained by electrochemical deposition by*  
395 *Tikhonov’s method//Nano-and microsystems technology*, R D Tikhonov , ? ? Cheremisinov , D V Gorelov ,  
396 V Ju . 2020 p. .
- 397 [Tikhonov et al. ()] R D Tikhonov , S A Polomoshnov , V V Amelichev , ? ? Cheremisinov , A M Kovalev .  
398 *CoNiFe triple system films formation by electrochemical deposition//Proc. Univ. Electronics*, 2021. 26 p. .
- 399 [Azizi et al. ()] *Tuning the Crystal Structure and Magnetic Properties of CoNiFeB Thin Films//Chemistry of*  
400 *Materials*, A Azizi , A Yourdkhani , D Cutting , N Pesika . 2013. 25 p. .